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WITNESS my hand this  
Twenty-eighth day of April 2004

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**Patents Act 1990**

**PROVISIONAL SPECIFICATION**

**Invention Title: Reductive Ammoniacal Leaching of Nickel and Cobalt Bearing Materials**

**Applicant: QNI Technology Pty Ltd.**

**The invention is described in the following statement:**

Reductive Ammoniacal leaching of Nickel and Cobalt bearing materials.

The invention provides an improved method of dissolution of nickel and cobalt materials in ammoniacal leaching systems by the addition of reducing agents.

- 5 More specifically, it describes an enhanced method of leaching a mixed nickel cobalt hydroxide precipitate in ammonia-ammonium carbonate solutions with the addition of a nickel, cobalt or nickel/cobalt sulphide or hydroxylamine as a reductant.

10 Background of the invention

- Mixed impure nickel/cobalt carbonates or hydroxides are often produced as intermediate products, as a step in the extraction of nickel and cobalt from their ores. These intermediates are then leached, selectively where possible to minimise impurities, and the resulting solutions treated in a variety of separation  
15 processes to produce refined nickel and refined cobalt. Leaching in ammoniacal solutions is well known and is in use commercially.

- A process of treating a mixed nickel/cobalt hydroxide precipitate produced from pressure acid leaching of laterite ore is described by Simmons et al. in  
20 Canadian Patent no. 618826, where the mixed hydroxide precipitate can be re-leached in ammonium hydroxide, ammonium carbonate or ammonium sulphate alone or in combination.

- The process developed for the Cawse Nickel project in Western Australia, was  
25 described in a paper presented at the Proceedings of the nickel/cobalt 97 International Symposium, 1997, Canada. The paper describes the process to extract nickel and cobalt from laterite ore by pressure acid leaching and describes the redissolution of the nickel and cobalt hydroxides produced as an intermediate by subjecting them to an ammonia leach, which it describes as  
30 "proven technology".

The use of ammoniacal solutions for re-leaching the mixed nickel/cobalt hydroxide has the advantage that it is selective in preferentially extracting nickel and cobalt, leaving behind impurities such as iron, manganese, and

magnesium. However it has the disadvantage that it does not extract all the nickel and cobalt from the hydroxides, leaving behind a residue that is rich in the two metals.

- 5 This does not constitute a major problem on a plant site where the ore is processed completely through to the final nickel and cobalt refined products in one location, as the nickel and cobalt rich residue from the re-leach step can be recycled to the initial ore treatment step to recover most of the metal values. An example of this is in commercial operation in Australia, where the high nickel  
10 and cobalt content residue from an inefficient leach of a mixed nickel/cobalt hydroxide in an ammonia ammonium carbonate solution is recycled to the primary leach stage to achieve an acceptable metal recovery efficiency.

- 15 However, mixed impure nickel/cobalt hydroxides may be shipped as concentrated intermediates for processing at another site, without facilities for recycling the residue. The mixed nickel and cobalt hydroxides also exhibit a tendency to "age" during the transport process caused by oxidation of the  $\text{Co}^{2+}$  to  $\text{Co}^{3+}$ , increasing the difficulty of dissolution, and increasing the amount of nickel and cobalt left in the leach residue. The incomplete dissolution of nickel  
20 and cobalt in the ammoniacal solution in these situations then necessitates a further expensive treatment step, or results in a loss of valuable product.

- The use of reductants to enhance metal recovery in ammoniacal leaching systems is reported in the literature and various schemes have been proposed  
25 to recover metal values (principally nickel, cobalt and copper) from manganese oxide ocean nodules.

- Steemson in a paper presented at the Alta nickel conference(1999) in Perth described the application of reductive leaching, using sulphur dioxide, for the  
30 recovery of nickel and cobalt from a mixed hydroxide in an ammonia/ammonium sulphate medium, as part of the Ramu project.

The prior art processes do not provide for a complete and economical recovery of nickel and cobalt from a mixed nickel/cobalt hydroxide.

Furthermore the inventors have identified two different ageing mechanisms operating when crude mixed nickel/cobalt hydroxides are stored for extended periods. The first ageing process is oxidative and primarily affects cobalt redissolution and is largely overcome by the use of a reductant in the ammoniacal leach liquor. The second "ageing" mechanism affects the nickel and appears to be associated with co-precipitated magnesium compounds.

An improvement on the prior art would therefore be to achieve a more complete dissolution and hence recovery of nickel and cobalt from a nickel/cobalt hydroxide during the leaching process in an ammonia ammonium carbonate solution, overcoming any aging effects of the material, while minimizing releaching of impurities, and minimising reagent costs. It is a desired feature of the present invention to provide a process that achieves improvements in the recovery of nickel and/or cobalt from nickel and/or cobalt hydroxide and remains commercially economic.

The discussion of the background to the invention herein is included to explain the context of the invention. This is not to be taken as an admission that any of the material referred to was published, known or part of the common general knowledge in Australia as at the priority date of any of the claims.

Throughout the description and claims of the specification the word "comprise" and variations of the word, such as "comprising" and "comprises", is not intended to exclude other additives, components, integers or steps.

#### Summary of the invention.

The present invention resides in a method of leaching nickel and cobalt from a mixed nickel/cobalt hydroxide material in an ammonia ammonium carbonate solution together with a reductant. The reductant is preferably selected from hydroxylamine, nickel sulphide, cobalt sulphide or a mixed nickel/cobalt sulphide.

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5 The preferred reductant is a mixed nickel/cobalt sulphide which can be produced as part of the reaction process. Preferably, a portion of the ammoniacal ammonium carbonate leach solution from a Caron type process containing some nickel and cobalt (that is the product liquor from leaching a reduced laterite ore with an ammonia ammonium carbonate solution) is treated  
10 with ammonium hydrosulphide or sodium hydrosulphide to produce a solid precipitate of mixed nickel/cobalt sulphide. This mixed nickel/cobalt sulphide is then used as the reductant together with ammonia ammoniacal carbonate leach solution to leach the nickel/cobalt hydroxide in accordance with the process of the present invention. Alternatively nickel sulphide or cobalt sulphide may be  
15 produced rather than a mixed nickel/cobalt sulphide, depending on the nickel and cobalt levels in the ammonium solution.

20 The process may include further leach steps to improve the recovery of the nickel and cobalt wherein at least one leach step is conducted with a combination of the ammonia ammonium carbonate leach solution and a reductant.

Accordingly, in a further embodiment, the present invention resides in a process for the recovery of nickel and/or cobalt from a mixed nickel/cobalt hydroxide material including the steps of:

- a) providing a mixed nickel/cobalt hydroxide material;
- b) contacting the hydroxide material with an ammonia ammonium carbonate leach solution in a primary leach step;
- c) separating the residue of the primary leach step from the product solution;
- 30 and
- d) contacting the residue with further ammonia ammonium carbonate leach solution and a reductant in a secondary leach step.

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4. Separating the residue from the secondary leach from the resultant product solution and then returning the product solution to the primary leach stage by mixing the product solution with the feed ammonia ammonium leach carbonate leach solution. The residue from the secondary leach step is subjected to prolonged contact with strong ammoniacal ammonium carbonate liquor to further enhance the recovery of nickel and cobalt. The residue following this prolonged contact with the ammoniacal liquor is separated and discarded while the enriched product solution is sent for further processing to separate the cobalt and the nickel.

It is possible, depending on the degree of "ageing" of the nickel/cobalt hydroxide feed, that the residue from the second leach stage will be small in mass and have little or no nickel and cobalt content and will not require a third leach stage. In this case the preferred leach process would have two leach stages, the second stage in the presence of the reductant.

A preferred embodiment of the process of the invention regardless of the number of leach steps, is the use of a mixed nickel/cobalt sulphide or nickel sulphide or cobalt sulphide as the reductant. Most preferably, a mixed nickel/cobalt sulphide is used as the reductant. A mixed nickel/cobalt sulphide has the advantage that it can be produced simply from ammoniacal solutions used in nickel and cobalt leach processes by the addition of ammonium hydrosulphide or sodium hydrosulphide. The production of nickel sulphide, cobalt sulphide or a mixed nickel/cobalt sulphide depends on the nickel and/or cobalt content in the ammonia ammonium carbonate leach solutions.

The product solution from leaching the hydroxide material as summarised above, or ammoniacal ammonia carbonate process liquors such as those

available in the Caron process are suitable for preparation of the mixed nickel/cobalt sulphide reductant in this manner.

5 Mixed nickel/cobalt sulphide is also significantly cheaper than hydroxylamine to use as a reductant and minimises the introduction of additional chemicals to the leach process.

10 Typically, the process of the present invention is useful in the nickel and cobalt recovery process where it is necessary to recover nickel and cobalt from reduced laterite ore by leaching in ammonia ammonium carbonate solution, such as the Caron process. It is contemplated that the process of the present invention may be integrated into such a process, where the metal values from a mixed nickel/cobalt hydroxide intermediate product are to be recovered into the process.

15 The process of the present invention is also useful in the nickel and cobalt recovery process where it is necessary to recover nickel and cobalt from an acid leach process from laterite ore. It is contemplated that the process of the present invention may be integrated into such a process, where nickel and cobalt are recovered as a mixed nickel/cobalt hydroxide product and redissolved in ammonia ammonium carbonate solutions. In such a process, the improved recovery of metal values using the present invention will minimize the recycling of nickel and cobalt to the ore treatment section, reducing processing cost and improving operational efficiencies.

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#### Detailed Description of the attached drawings

30 A nickel and cobalt recovery process integrating the process of the present invention is described by reference to Figure 1, which is a flowsheet of the single stage leach and Figure 2 which is a flowsheet describing the two and three stage leach. Both flowsheets describe the use of a mixed nickel/cobalt sulphide as a reductant in the leaching process.

The process described in Figure 1 illustrates the application of the single stage reductive leach to the Caron process. Reduced laterite nickel ore (1) is leached by an ammonia ammonium carbonate leach solution (2) in the ore leach plant to produce an ore leaching (3) and a product liquor (4). The product liquor is the ammonia ammonium carbonate leach solution of the process of the present invention. The ammonia ammonium carbonate leach solution contains typically 8-16% ammonia, 4-12% carbon dioxide, 0.5-1.5% nickel and 0.02-0.2% cobalt.

A portion of the ammonia ammonium carbonate leach solution containing some nickel and cobalt, is treated with ammonium hydrosulphide (or sodium hydrosulphide) to produce a solid precipitate of mixed nickel/cobalt sulphide (5). The mixed sulphide is added, together with the mixed nickel/cobalt hydroxide feed (7), and the ammonia ammonium carbonate leach solution, being a portion of the ore leach product liquor. The mixture is agitated, together with air or oxygen containing gas injection, for a residence time of between thirty minutes and twelve hours.

The nickel and cobalt from both the mixed hydroxide and the mixed sulphide are leached into the ammonia ammonium carbonate leach solution, together with some minor impurities in the form of manganese, magnesium and iron from the mixed hydroxide. The enriched product liquor (25) is separated from the residue (8) and added to the remaining product liquor which proceeds for further processing to separate the nickel and cobalt (9) and extract them separately from solution.

The enriched product liquor may have a nickel content of between 1 gram per litre and 30 gram per litre, depending on the nature of the downstream processing required.

The leach residue is rejected or recycled to the ore treatment section where it is possible to do so.

The process described in Figure 2 illustrates the application of the two or three stage reductive leach to the Caron process. In this process the major part of the

nickel and cobalt present in the mixed hydroxide is dissolved in a primary leach stage, leaving a smaller quantity of material for the secondary leach stage, which then requires a smaller quantity of the mixed sulphide reductant while still allowing a higher reductant ratio to the metals to be dissolved. The leach solution used in the second stage leach also contains very low levels of nickel and cobalt, further improving the dissolution process for the metals.

Laterite nickel ore (10) is leached by an ammonia ammonium carbonate leach solution (11) in the ore leach plant to produce ore leaching (12) and a product liquor solution (13). The product liquor is the ammonia ammonium carbonate leach solution of the process of the present invention. The ammonia ammonium carbonate leach solution contains typically 8-16% ammonia, 4-12% carbon dioxide, 0-1.5 % Nickel and 0-0.2 % cobalt, preferably 0-1.0% nickel and 0.01% cobalt.

The mixed nickel/cobalt hydroxide feed (17), is added to a mixture of a portion of the product liquor solution (13) and the secondary leach product liquor (22) (when available) and agitated together in the primary leach stage for a residence time of between thirty minutes and twelve hours to obtain maximum dissolution of the nickel and cobalt values.

The residue from the primary leach stage (19) is separated from the enriched product liquor (18) and passes to the secondary leach stage.

A portion of the product liquor solution (13) is treated with ammonium hydrosulphide (or sodium hydrosulphide) to produce a solid precipitate of mixed nickel and cobalt sulphide (14). This is transferred as the reductant (15) to the secondary leach stage where it is mixed with the primary leach residue and a portion of the ammonia ammonium carbonate leach solution (11) that is used for ore leaching. This solution contains only trace values of nickel and cobalt, as it generally has not been used in the ore leaching step, which assists, together with the presence of the reductant, to obtain maximum dissolution of both nickel and cobalt. The mixture is aerated with air or oxygen containing gas during the secondary leaching stage, which has a residence time of thirty minutes to twelve hours. The remaining nickel and cobalt values are efficiently extracted in

the secondary leach into the secondary leach product liquor (22), which is separated from the secondary leach residue (21) and returned to the primary leach.

- 5 The enriched product liquor (18) from the primary leach and the enriched product liquor (22) from the secondary leach, are added to the remaining product liquor (13) which proceeds for further processing to separate the nickel and cobalt (24) and extract them separately from solution.
- 10 The enriched product liquor may have a nickel content of between 1 gram per litre and 30 gram per litre, depending on the nature of the downstream processing required.
- 15 The secondary leach residue is either returned to the roasting and leaching stage of the plant, rejected or recycled to the third leach stage as required. In the third leach stage, the residue is contacted in a soak of strong ammoniacal ammonia carbonate leach solution to recover further nickel and cobalt in a product liquor, which then proceeds for further processing to recover the nickel and cobalt. The residue will generally be sent for disposal.

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#### Examples

The following examples demonstrate the effectiveness of the invention.

- 25 Tests have been conducted with hydroxylamine sulphate which is commonly used as a reductant, to illustrate the concept of reductive leaching in ammoniacal solutions.

- 30 Table 1 shows the dissolution from a commercially produced mixed Nickel/Cobalt hydroxide, in an ammonia-ammonium carbonate solution, with and without hydroxylamine sulphate addition.

**Table 1. Effect of Hydroxylamine Addition on Hydroxide Leaching**

	Feed Mixed Hydroxide	Hydroxylamine Leach Residue	Control Leach Residue with no reductant
Solids Composition			
% Ni	37.2	2.2	10.3
% Co	3.8	0.46	2.9
% Mn	6.8	35.5	35.5
Ni dissolution (%)		99	96
Co dissolution (%)		98	80

Lixiviant composition : 93 g/L  $\text{NH}_3$ , 68 g/L  $\text{CO}_2$

Leach temperature : 30°C

5 Leach time : 45 minutes

Solids : equivalent to 10 g dry hydroxide (4 g (Ni + Co) /500 mL lixiviant

Hydroxylamine dosage : 4.50g of  $(\text{NH}_2\text{OH})_2 \cdot \text{H}_2\text{SO}_4$ /test

10 Table 2 shows the dissolution from a mixed Nickel cobalt hydroxide leach residue, after two prior stages of leaching in ammonia-ammonium carbonate liquors.

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**Table 2. Effect of Hydroxylamine addition on Releach of Hydroxide Leach Residue**

	Feed	After two initial leaches	After reductive releach	After control releach with no reductant
Lixiviant		Caron product liquor, followed by ore leach solution	Caron Ore leach solution	Caron Ore leach solution
Leach Time (min)			180	180
Leach Temp (°C)			90	90
<b>Solids Composition</b>				
% Ni	45.4	7.32	3.74	5.38
% Co	1.62	0.97	0.54	0.77
% Mn	3.14	12.9	13.0	13.7
% Mg	3.00	13.9	13.9	14.1
<b>Incremental Dissolution</b>				
Ni (%)		95.6	50.9	35.4
Co (%)		82.4	46.0	27.8
Mn (%)		6.7	0.3	0.1
<b>Cumulative Dissolution</b>				
Ni (%)		95.6	97.8	97.2
Co (%)		82.4	90.5	87.3
Mn (%)		6.7	7.0	6.8

Lixiviant composition : 155 g/L  $\text{NH}_3$ , 105 g/L  $\text{CO}_2$

5 Leach time : 180 minutes

Solids : 10% w/w (122 g residue (dry basis)/test

Hydroxylamine dosage : 56.2 of  $(\text{NH}_2\text{OH})_2\cdot\text{H}_2\text{SO}_4$ /test

10 Table 1 demonstrates that the addition of hydroxylamine improved the Nickel and Cobalt dissolution from the mixed hydroxide. Table 2 demonstrates the applicability of the reductant addition to releaching of mixed hydroxide leach residues. This offers an alternative to reprocessing of leach residues via the normal ore processing route.

Results from leaching testwork without reductant on the commercial sample indicated that the dissolution of cobalt from the mixed hydroxide declined with age of the hydroxide, in agreement with leachability tests on other hydroxides (known as the ageing effect). Addition of reductant (hydroxylamine sulphate) to the leach test overcame the ageing effect.

In the following example, a mixed nickel cobalt sulphide was produced by treating a typical 'Caron' process product liquor with ammonium hydrosulphide for the leaching of either mixed nickel cobalt hydroxide in a single stage leach, or the residue from a previous ammoniacal leach of mixed hydroxide in the two stage leach.

Three cases have been tested:

1. a single stage leach in Product Liquor with sulphide as a reductant,
2. a primary leach in Product Liquor (without sulphide as a reductant, and no aeration), followed by a secondary leach of the first stage residue with the mixed sulphide as a reductant, or
3. a tertiary leach/soak in strong ammoniacal carbonate solution.

In either the single stage leach with sulphide (case 1), or the two stage leach with sulphide (case 2), the sulphide leach consisted of a 30 minute anaerobic leach, followed by an aerobic leach (the latter achieved by oxygen sparging at 0.25 v/v oxygen at atmospheric pressure for 120 minutes, or pressurising with oxygen to 500 kPag for 30 minutes in a laboratory autoclave).

The initial mixed hydroxide was from the commercial supply, and 6 - 8 months old at the time of testing.



Results are shown in Tables 3 and 4.

**Table 3. Single Stage Leaching of Mixed hydroxide with Mixed Sulphide reductant**

Pressure Test		Oxygen (atmospheric)		500 kPag Oxygen	
		No reductant	Reductant	No reductant	Reductant
Mixed sulphide (%w/w) (see note a. below)		0	0.12	0	0.12
Mixed hydroxide (%w/w)		2.9	2.9	2.6	2.8
Lixiviant-Caron product liquor/ore leach solution					
Anaerobic Leach (min)		30	30	30	30
Aerobic Leach (min)		120	120	30	30
Leach Temperature (°C)		65	65	80	80
Solids	Initial MHP	Primary Residue	Primary Residue	Primary Residue	Primary Residue
Ni (%)	38.8	8.19	2.49	8.23	2.12
Co (%)	4.10	3.07	1.10	2.73	1.52
Mn (%)	6.91	32.30	32.08	30.27	32.5
Total Dissolution					
Ni (%)		95.1	98.6	95.8	98.8
Co (%)		82.9	95.4	87.1	92.6
Dissolution from MHP					
Ni (%)		95.1	98.5	95.6	98.8
Co (%)		82.9	94.1	97.1	90.6

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a. Reductant solids composition 17% Ni, 29% Co, 29% S.

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**Table 4. Second Stage Reductive Leaching of Primary Leach Residue with Mixed Sulphide**

Pressure	Test	O <sub>2</sub> (atmospheric)		500 kPag O <sub>2</sub>	
		Control-no reductant	Reductant	Control-no reductant	Reductant
Mixed sulphide (%w/w)		0	0.44	0	0.44
Mixed Hydroxide (%w/w)		2.1	2.0	2.1	2.0
Lixiviant	Caron Product liquor	Ore leach solution	Ore leach solution	Ore leach solution	Ore leach solution
Anaerobic Leach (min)		30	30	30	30
Aerobic Leach (min)		120	120	30	30
Leach Temperature (°C)		65	65	80	80
Solids	Initial MHP Primary Residue	Secondary Residue	Secondary Residue	Secondary Residue	Secondary Residue
Ni (%)	8.56	7.37	1.92	6.48	1.44
Co (%)	2.70	2.17	1.02	2.29	0.62
Mn (%)	30.34	29.70	31.77	30.82	31.95
<b>Total Dissolution</b>					
Ni (%)	95.5	-5.7	80.0	16.3	86.9
Co (%)	88.7	-9.6	85.8	6.3	92.4
<b>Dissolution from Mixed Hydroxide First stage Residue</b>					
Ni (%)		-5.7	72.0	16.3	81.5
Co (%)		-9.6	54.6	6.3	75.7
<b>Cumulative Dissolution from original Mixed hydroxide feed</b>					
Ni (%)		95.5	98.7	96.2	99.2
Co (%)		88.7	94.0	87.5	96.8

- 5 Original Mixed Hydroxide composition was as shown in Table 3.

The results in Tables 3 and 4 indicate that the dissolution from either mixed hydroxide or primary leach residue was improved by the addition of sulphide. Moreover, it was demonstrated that the high total recoveries from the hydroxide

(98 – 99% for Ni and 94% or better for Co) could be obtained by the use of a reductant. Ordinarily, it would be expected that ageing effects on this hydroxide would have meant that the maximum Nickel and Cobalt dissolutions would have been about 96% and 90%, respectively, on material that was up to 3 months old. The same material, after six months, would probably have shown similar dissolutions as that observed after three months.

The result in Table 5 shows the affect of the tertiary leach having strong ammonium carbonate liquor to treat the residue from the two stage reductive leach.

**Table 5**

**Cumulative  
% Recovery**

<b>Solids</b>	<b>Ni</b>	<b>Co</b>
Secondary residue	97.8	95.2
Tertiary residue	99.0	97.6

The process of the present invention, which preferably includes either a primary, secondary or tertiary stage reductive leach to dissolve a mixed nickel/cobalt hydroxide precipitate in ammoniacal solutions, using hydroxylamine or a mixed nickel cobalt sulphide as the reductant, has several advantages over the conventional oxidative leach process for the recovery of nickel and cobalt.

A particular advantage is that the recovery into solution of nickel and cobalt is significantly higher using the reductive leach process. This is particularly important where the mixed hydroxide is to be processed in a location remote from the original ore processing step, where the leach residue containing unrecovered nickel and cobalt therefore can not be reprocessed with the ore feed. It is also useful where the mixed hydroxide is to be processed in situ, as the reductive leach minimises the quantity of residue which may be recycled to ore processing, hence minimising process inefficiencies and costs.

A further advantage is that the mixed nickel cobalt sulphide used as the reductant is particularly easy to produce in the process, and is amenable to re absorption into the process, as it contains the two metals which are to be recovered. This is particularly useful where the process receiving the mixed hydroxide is the Caron ammonia ammonium carbonate process, where a mixed nickel cobalt sulphide is easily produced, typically by the addition of ammonium hydrosulphide or sodium hydrosulphide to any of the process streams containing nickel and cobalt.

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A further advantage is that the dissolution process eliminates the impurities such as manganese, magnesium, and iron that may be present in the mixed hydroxide intermediate, as they are eliminated from the enriched product liquor solution.

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A further advantage, which is particularly useful in the Caron process, is that the production of the mixed nickel/cobalt sulphide reductant by treatment of a process stream from the ammonia ammonium carbonate leach circuit with ammonium hydrosulphide, has the effect of depleting the cobalt content of the process stream, which improves the leaching of cobalt in the ore leach stage, and increases the overall cobalt recovery of the process.

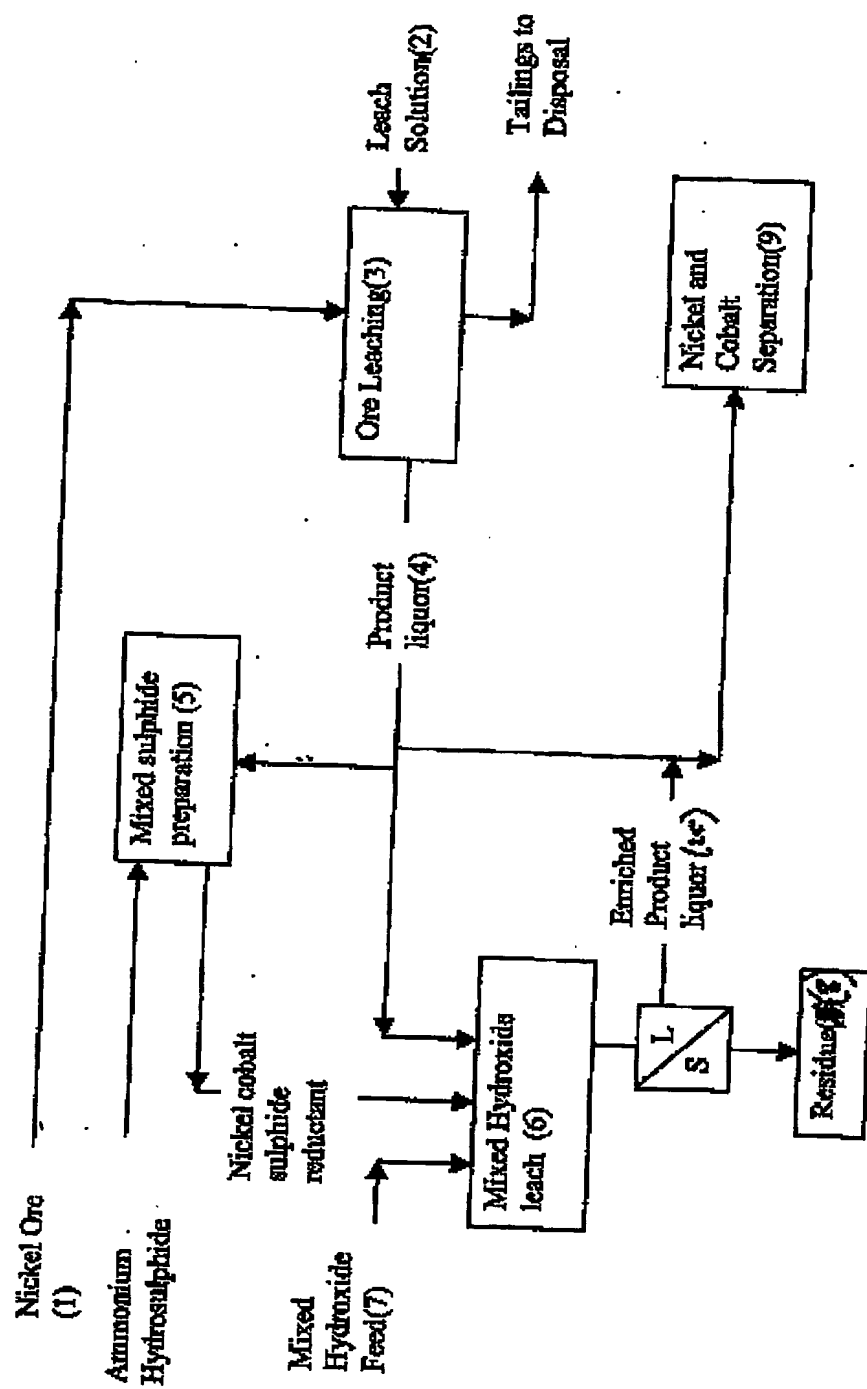
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The above description is intended to be illustrative of the preferred embodiments of the present invention. It should be understood by those skilled in the art that many variations or alterations may be made without departing from the spirit of the invention.

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30 DATED: 11 April 2003  
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**Fig 1 Single stage Reductive Leach Process**

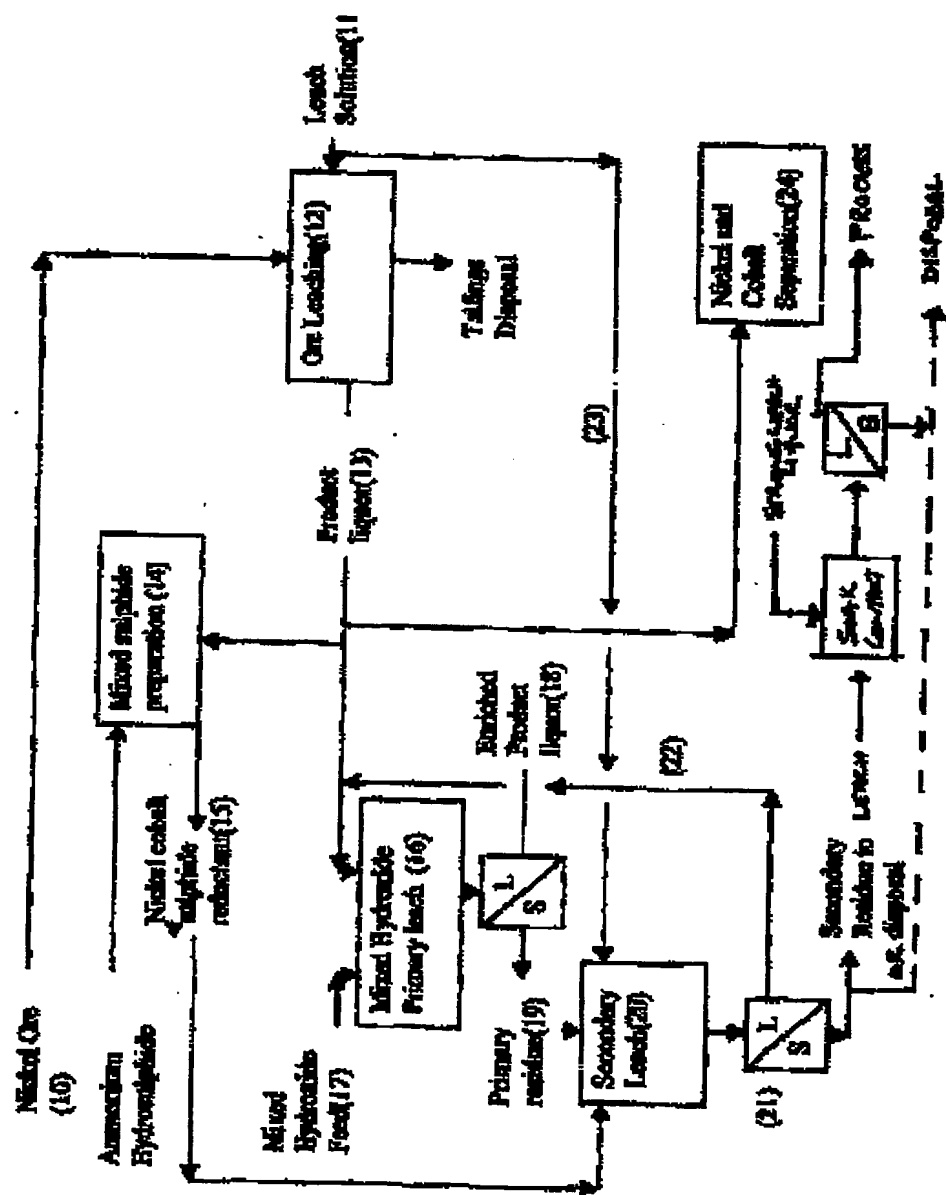


Fig 2 Two Stage Reductive Leaching Process

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